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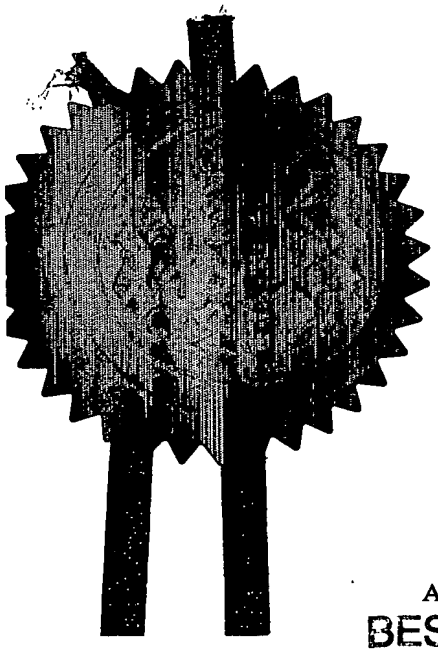

Signed J e Behen

Dated 23 July 2003

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17JUL02 E733729-1 D00518
P01/7700 0.00-0216502.5

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(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

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1. Your refere — KJL/CT/P5049

2. Patent application numb — 0216502.5
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17 JUL 2002

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Epichem Limited
Power Road
Bromborough
Wirral
Merseyside L62 1TZ
United Kingdom

Patents ADP number *(if you know it)* 6094668003

If the applicant is a corporate body, give the country/state of its incorporation

4. Title of the invention

A method and apparatus for monitoring liquid levels within a vessel

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

ROYSTONS,

Tower Building,
Water Street,
Liverpool. L3 1BA
Merseyside.

Patents ADP number *(if you know it)* 1438001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

Yes

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form	7
Description	1
Claim(s)	-
Abstract	-
Drawing(s)	2 only

CF

10. If you are also filing any of the following, state how many against each item.

Priority documents	-
Translations of priority documents	-
Statement of inventorship and right to grant of a patent *(Patents Form 7/77)*	-
Request for preliminary examination and search *(Patents Form 9/77)*	-
Request for substantive examination *(Patents Form 10/77)*	-
Any other documents *(please specify)*	-

11. I/We request the grant of a patent on the basis of this applicatio

Signature ROYSTONS Royston Date 17/07/02

12. Name and daytime telephone number of person to contact in the United Kingdom K.J. Lees - 0151-236 5147/1417

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Notes

a) *If you need help to fill in this form or you have any questions, please contact the Patent Office on 08459 500505.*

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d) *If you have answered 'Yes' Patents Form 7/77 will need to be filed.*

e) *Once you have filled in the form you must remember to sign and date it.*

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Title: A Method and Apparatus for monitoring liquid levels within a vessel.

DESCRIPTION

The present invention relates to the field of measuring probes, particularly but not exclusively a probe for monitoring the level of a reactive liquid, such as an organometallic compound, within a vessel.

Thin films of metal are fabricated upon solid substrates for use in the electronics and opto-electronics industries by a process known as Metal Organic Chemical Vapour Deposition (MOCVD). The metal precursors are generally delivered to a substrate by means of a bubbler. A bubbler containing the metal precursor material has an inlet pipe, a dip-tube and an outlet pipe. Carrier gas, such as hydrogen, helium or nitrogen, is introduced into the vessel through the inlet pipe and delivered to the bottom of the vessel though the dip-tube. The gas is able to pick up some of the vapours of the metal precursor before leaving the bubbler via the outlet pipe. The gas transports the precursor vapour to a reactor site where the precursor is decomposed to provide a deposit of the metal on a solid substrate.

The amount of precursor material in the bubbler is constantly changing with use of the apparatus. Conventional probes cannot be used due to the organometallic compounds attacking the probe. This would also lead to contamination of the precursor. The volume of precursor in the bubbler may be measured using single point measurements for high and low fill levels based on an optical system. However, this utilizes a glass tube which may break causing contamination of the contents of the bubbler. Alternatively, the bubbler may be provided without an accurate level

indicator, being changed either when a drop in performance is seen or using a calculated figure based on usage time under particular conditions. This can lead to early replacement of the bubbler when potentially another 10% of material in the bubbler could be used.

Clearly it would be desirable to provide an accurate probe for monitoring the level of organometallic precursor contained within a bubbler. However, problems arise with the provision of a suitable probe that has the necessary robustness and compatibility with the organometallic compounds with which it comes into contact. In this respect, the probe must not react with the chemicals contained within the vessel to ensure high purity metal deposits are achieved. Additionally, it should not impair the airtight seal of the bubbler since the contents are air-sensitive and should not affect the pressure within the vessel.

It is an object of the present invention to provide a method of continuously monitoring the level of a reactive liquid in a vessel, particularly but not exclusively the level of an organometallic compound, which aims to overcome, or at least alleviate, the abovementioned drawbacks.

A further object of the present invention is to provide an apparatus for continuously monitoring the level of a liquid in a vessel, particularly but not exclusively an organometallic compound, that aims to overcome, or at least alleviate, the abovementioned drawbacks.

Accordingly a first aspect of the present invention provides a method for monitoring the level of a liquid in a vessel, the method comprising the steps of inserting at least one metallic probe coated with an elastomeric material into a vessel to act as a first electrode, hermetically sealing the probe within the vessel, providing a

further electrode spaced apart from the first electrode to form a capacitor, applying an electric current to the capacitor and monitoring the capacitance thereof.

A second aspect of the present invention provides an apparatus for monitoring the level of a liquid in a vessel, the apparatus comprising at least one metallic probe hermetically sealed within the vessel to act a first electrode, the metallic probe having a coating of an elastomeric material over at least the part of the probe that extends from the seal, a second electrode spaced apart from said first electrode to form a capacitor, means for applying an electric current to the capacitor and means for monitoring the capacitance thereof.

It is to be appreciated that the two spaced apart electrodes are provided in order to set up the necessary dielectric therebetween. The capacitance of the capacitor thus formed will vary with the amount of liquid between the probes thereby enabling the level of liquid within the vessel to be monitored.

Preferably the vessel itself is a metallic container, for example being of stainless steel, and acts as the second electrode. Alternatively, two parallel probes could be sealed within the apparatus to act as the first and second electrodes respectively.

The metallic probe that is inserted into the vessel is preferably in the form of a rod or flat elongated plate. The probe is particularly suitable for use in monitoring organometallic compounds. The probe is preferably attached to a port at the top of a vessel, the vessel generally being in the form of a bubbler, that contains an inlet and an outlet pipe. The probe is preferably encased in a glass material at one end thereof and this is hermetically sealed within the port.

Conventional means may be provided for applying an AC or DC source to the probe, together with monitoring means, such as a capacitance meter, for measuring a change in capacitance. Preferably, a recorder is also provided for recording the change in capacitance. The recorder may include display means, such as a liquid crystal display. It is preferable for the capacitance to be continuously monitored thereby providing a continuous reading of the level of liquid in the vessel.

Preferably, the apparatus includes means for calibration of the system whereby a particular capacitance corresponds to a particular volume of liquid within the vessel. For example, the recording means could be set at a value of "0" for a capacitance recorded for an empty vessel and could be set "100" for the capacitance recorded for a full vessel. Preferably, the apparatus is calibrated to respond to particular characteristics of the liquid contained within the vessel. Additionally, the apparatus may be adapted to provide the rate of removal or addition of the liquid to the vessel.

In a preferred embodiment of the present invention there is provided a bubbler containing an organometallic compound, the bubbler comprising a sealed metallic container having an inlet pipe, an outlet pipe and a dip-tube and further comprising a metallic probe hermetically sealed within the container, the metallic probe having a coating of an elastomeric material over at least the part thereof that extends from the seal, the container and the probe forming a capacitor, means for applying an electric current to the capacitor and monitoring means for measuring the capacitance thereof.

For a better understanding of the present invention and to show more clearly how it may be carried into effect reference will now be made to the following Examples in which Example 1 investigates the use of an apparatus according to the present invention in measuring the changing level of trimethylgallium (TMG) in a

bubbler and Example 2 investigates the use of an apparatus according to the present invention in measuring the changing level of trimethylaluminium (TMA) in a bubbler, and with reference to the accompanying drawings in which:-

Figure 1 is a schematic diagram of an apparatus fitted with a probe according to one embodiment of the present invention;

Figure 2 is a cross-sectional view through the probe contained in the apparatus of Figure 1;

Figure 3 is a graph showing the changing level of TMG in a bubbler with time measured using the apparatus of the present invention; and

Figure 4 is a graph showing the changing level of TMA in a bubbler with time measured using the apparatus of the present invention.

Referring to Figures 1 and 2 of the accompanying drawings, an apparatus for continuously monitoring the level of an organometallic compound in a vessel, such as a bubbler, is illustrated. The bubbler 2 has an inlet pipe 4 leading into a dip pipe 6 and an outlet pipe 8. A metal probe 10 is provided which extends through the centre and length of the bubbler vessel. The upper end of the probe is encapsulated in a layer of glass 12 and hermetically sealed to the top of the bubbler to ensure that the pressure within the bubbler is maintained and that the contents of the bubbler are isolated from air. The lower end of the probe that dips into the precursor contained within the vessel is coated with an inert, heat resistant material, such as polytetrafluoroethylene (PTFE) or other elastomeric material. The top of the probe is connected to an electronic control unit which includes a power source and communicates with a monitoring device that displays a reading of the level of the liquid in the bubbler.

Example 1

An investigation was carried out to demonstrate the ability of an apparatus according to one embodiment of the present invention to continuously monitor the level of trimethylgallium (TMG) in a bubbler. A probe as hereinbefore described was sealed within a bubbler and the monitor was zeroed when the bubbler was empty. The bubbler was then filled with TMG (ca. 180g = 157ml) and the readings set to a maximum. The material was then pushed out of the bubbler through the dip leg and into a receiver. The reading level on the Teletrend™ was continuously recorded. The recording showed that as the material was removed the reading went from 100% to 0%, as illustrated in Figure 3 of the accompanying drawings. The steepness of the slope of the graph was dependent upon the rate of material removal. Once all the material had been removed, the system was checked by dropping more material into the bubbler wherein the reading rose from 0% back to 100%.

Example 2.

An investigation was carried out to demonstrate the ability of an apparatus according to one embodiment of the present invention to continuously monitor the level of trimethylaluminium (TMA) in a bubbler. The monitor connected to the probe was zeroed when the bubbler was empty as described above in relation to Example 1. It was then filled with TMA (ca. 120g = 160ml) and the readings set to a maximum. The material was then pushed out of the bubbler through the dip leg and into a receiver. The reading level on the Teletrend™ was recorded. The recording demonstrated that as the material was removed the reading went from 100% to 0%, as illustrated in Figure 4 of the accompanying drawings. The steepness of the slope was dependent upon the rate of material removal.

The present invention provides a measuring probe that is suitable for monitoring the level of materials in a bubbler that is robust and compatible with the organometallic compounds that are contained within the vessel. This enables the level of organometallic compounds in individual bubblers to be continuously monitored and allows customers to know when to order more material as the level of material in the bubbler is used up. This also ensures maximum utilisation of the source material within the bubbler. The glass haematic seal that connects the probe to the port of the bubbler ensures that the pressure within the vessel is retained and that the contents of the vessel, which are extremely air sensitive, remain isolated from air.

Additionally, the seal is resistant to high temperatures and does not interfere with the measurement of the level or with the content of the vessel. The elastomeric material which coats the probe provides total inertness to ensure that the purity of the precursor is maintained. Furthermore, the probe is less fragile than the glass probes previously provided. This is important because of rough transport handling. It is essential that the probes should not break since, due to the nature of the products inside the bubbler, any breakage could lead to a leak and possibly fire.

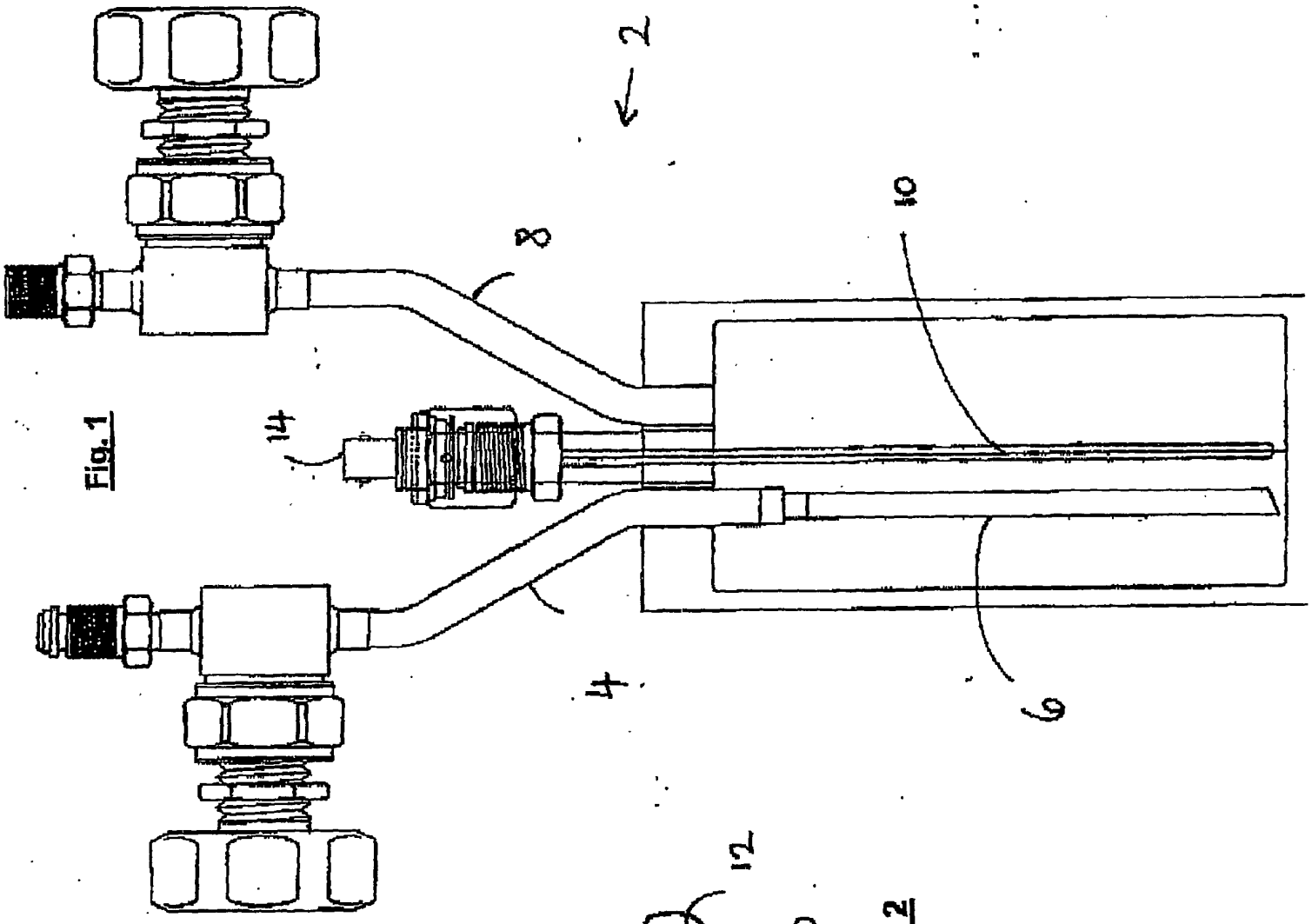
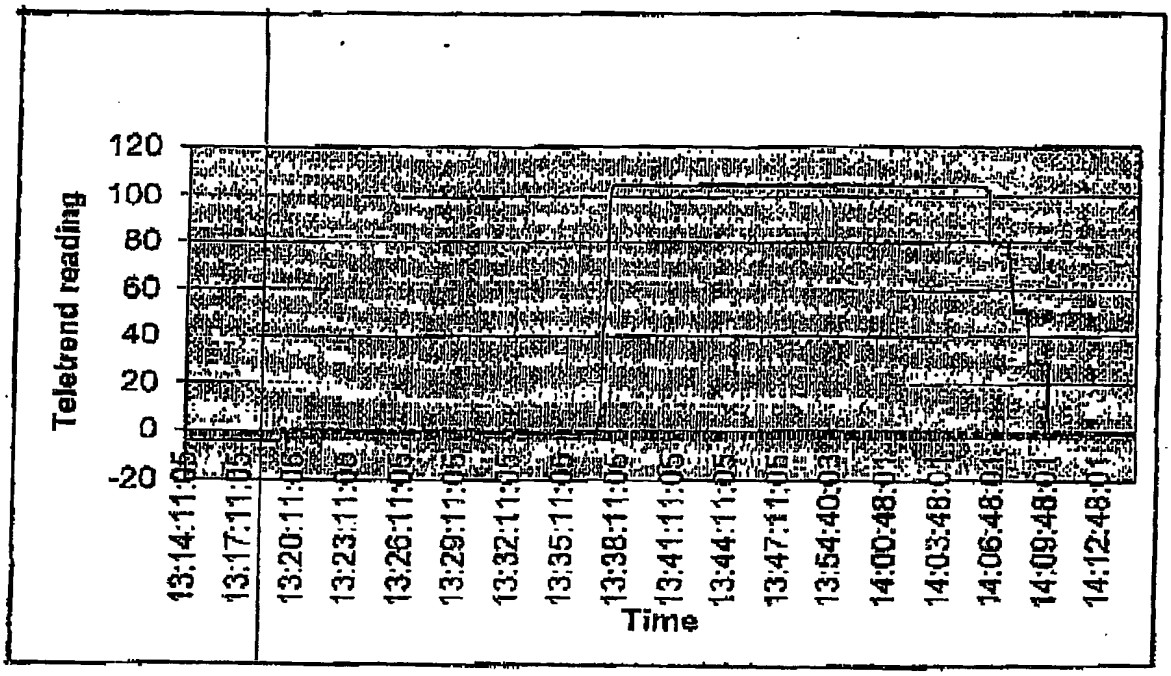


Fig.1

Fig.2

Fig. 3


Teletrend reading
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40
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-20
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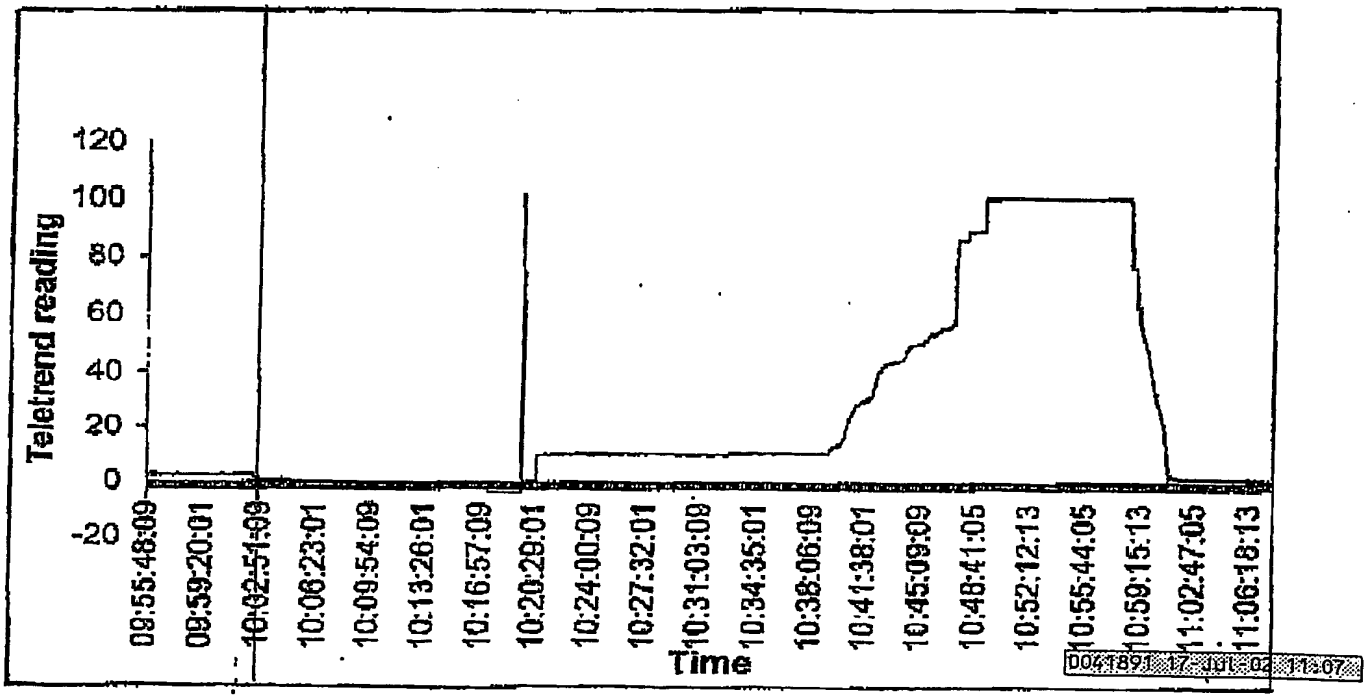
Time

Fig. 4


Teletrend reading
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10:02:51:09
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Time